इंटरनेट मानक

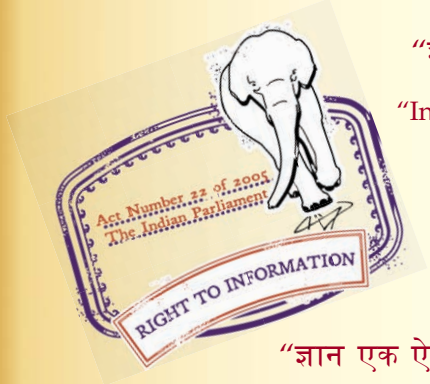
Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

"जानने का अधिकार, जीने का अधिकार"
Mazdoor Kisan Shakti Sangathan
"The Right to Information, The Right to Live"

"पुराने को छोड नये के तरफ"
Jawaharlal Nehru
"Step Out From the Old to the New"

IS 10369 (1982): Ethion, Technical [FAD 1: Pesticides and Pesticides Residue Analysis]



"ज्ञान से एक नये भारत का निर्माण"
Satyanarayan Gangaram Pitroda
"Invent a New India Using Knowledge"

"ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है"
Bhartṛhari—Nītiśatakam
"Knowledge is such a treasure which cannot be stolen"

BLANK PAGE

IS : 10369 - 1982
(Reaffirmed 2004)

Indian Standard

SPECIFICATION FOR ETHION, TECHNICAL

UDC 632.951 ETHION

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 3

May 1983

Indian Standard

SPECIFICATION FOR ETHION, TECHNICAL

Pest Control Sectional Committee, AFCDC 6

Chairman	*Representing*
DR K. D. PAHARIA	Directorate of Plant Protection, Quarantine & Storage (Ministry of Agriculture), Faridabad
Members	
DR H. L. BAMI	Central Forensic Science Laboratory (Central Bureau of Investigation), New Delhi
DR B. BANERJEE	Tea Research Association, Calcutta
DR G. SATYANARAYANA (*Alternate*)	
DR N. K. BASU	The Alkali & Chemical Corporation of India Limited, Calcutta
SHRI S. R. NENE (*Alternate*)	
DR A. K. BHATNAGAR	Hindustan Insecticides Limited, New Delhi
SHRI C. C. ABRAHAM (*Alternate*)	
CHIEF CHEMIST	Central Revenues Control Laboratory (Ministry of Finance), New Delhi
SHRI S. KRISHNAMURTHY (*Alternate*)	
CHIEF PLANT PROTECTION OFFICER	Department of Agriculture, Government of Maharashtra, Bombay
CHEMIST INCHARGE, INSECTICIDES TESTING LABORATORY (*Alternate*)	
SHRI V. G. DESHPANDE	Ciba-Geigy of India Limited, Bombay
SHRI E. A. ALMEIDA (*Alternate*)	
SHRI OM P. DHAMIJA	Export Inspection Council of India, New Delhi
SHRI P. P. RANGA RAO (*Alternate*)	
DR M. S. DHATT	National Malaria Eradication Programme, Delhi
DIRECTOR GENERAL	Directorate General Armed Forces Medical Services (Ministry of Defence), New Delhi
DIRECTOR OF AGRICULTURE	Department of Agriculture, Government of Uttar Pradesh, Lucknow
DEPUTY DIRECTOR (PLANT PROTECTION) (*Alternate*)	
SHRI V. DORAIRAJ	Plastic Containers Sectional Committee, MCPD 11, ISI
DRUGS CONTROLLER	Indian Pharmacopoeia Committee (Ministry of Health and Family Welfare), New Delhi
SHRI M. RAVIKANT (*Alternate*)	

(*Continued on page 2*)

(*Continued from page* 1)

Members	*Representing*
SHRI G. D. GOKHALE	Bombay Chemicals Private Limited, Bombay
SHRI V. V. KETKAR (*Alternate*)	
SHRI D. K. JAIN	Punjab United Pesticides and Chemicals Ltd, Chandigarh
JOINT DIRECTOR OF AGRICULTURE, (DEVELOPMENT & ICP)	Department of Agriculture, Government of Karnataka, Mysore
DEPUTY DIRECTOR OF AGRICULTURE (PLANT PROTECTION) (*Alternate*)	
DR M. G. JOTWANI	Entomology Division, Indian Agricultural Research Institute (ICAR), New Delhi
DR R. L. KALRA	Punjab Agricultural University, Ludhiana
DR R. P. CHAWLA (*Alternate*)	
DR KALYAN SINGH	Chander Sekhar Azad University of Agriculture & Technology, Kanpur
DR KRISHAN SINGH	Indian Institute of Sugarcane Research (ICAR), Lucknow
DR S. C. SRIVASTAVA (*Alternate*)	
DR K. KRISHNAMURTHY	Ministry of Agriculture (Department of Food), New Delhi
SHRI G. N. BHARDWAJ (*Alternate*)	
SHRI S. G. KRISHNAN	National Organic Chemical Industries Limited, Bombay
DR J. S. VERMA (*Alternate*)	
DR V. LAKSHMINARAYANA	Directorate of Plant Protection, Quarantine & Storage (Ministry of Agriculture), Faridabad
SHRI V. C. BHARGAVA (*Alternate*)	
SHRI S. K. LUTHRA	Directorate General of Technical Development, New Delhi
SHRI S. C. BAJAJ (*Alternate*)	
DR J. C. MAJUMDAR	BASF India Limited, New Delhi
DR B. P. CHANDRASEKHAR (*Alternate*)	
SHRI S. K. MAJUMDAR	Central Food Technological Research Institute (CSIR), Mysore
SHRI M. MUTHU (*Alternate*)	
SHRI O. N. MEHROTRA	Ministry of Defence (R & D)
SHRI M. G. CHATTERJEE (*Alternate*)	
SHRI K. S. MEHTA	Bharat Pulverising Mills Private Ltd, Bombay
SHRI S. CHATTERJI (*Alternate*)	
SHRI L. S. MIRLE	Agromore Limited, Bangalore
SHRI S. K. RAMAN (*Alternate*)	
SHRI J. M. MODI	Pesticides Formulators Association of India (S.S.I.), Bombay
DR S. R. BAROOAH (*Alternate*)	
DR A. L. MOOKERJEE	Cyanamid India Limited, Bombay
SHRI J. P. PARIKH (*Alternate*)	
DR S. K. MUKERJEE	Agricultural Chemicals Division, Indian Agricultural Research Institute (ICAR), New Delhi
DR N. K. ROY (*Alternate*)	
SHRI K. R. NARAYANA RAO	Food Corporation of India, New Delhi
SHRIMATI K. K. M. BHAVNANI (*Alternate*)	

(*Continued on page* 10)

AMENDMENT NO. 1 JANUARY 2007
TO
IS 10369 : 1982 SPECIFICATION FOR ETHION, TECHNICAL

(*Page* 4, *clause* 3.1) — Substitute 'The material shall be packed as per requirements given in IS 8190 (Part 2) : 1988*' *for* 'The material shall be packed as per requirements given in IS : 8190 (Part II) - 1980*' as applicable to packing of malathion, technical.'

(*Page* 4, *footnote marked**) — Substitute 'Requirements for packing of pesticides: Part 2 Liquid pesticides (*second revision*)' for the existing.

(*Page* 5, *clause* 4.1) — Substitute 'Representative samples of the material shall be drawn as prescribed in IS 10946 : 1996†' *for* 'Representative samples of the material shall be drawn as prescribed in 'Indian Standard methods for sampling of pesticides and their formulations' (*under preparation*).

(*Page* 5, *clause* 4.1, *Note*) — Delete Note and its contents.

(*Page* 5) — Insert the following footnote at the end of page:

'†Methods of sampling for technical grade pesticides.'

(FAD 1)

Reprography Unit, BIS, New Delhi, India

Indian Standard

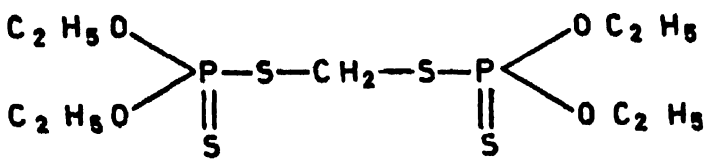
SPECIFICATION FOR ETHION, TECHNICAL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 10 November 1982, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Ethion, technical is employed in the preparation of number of insecticidal and acaricidal formulations.

0.3 Ethion is the accepted common name by the International Organization for Standardization (ISO) for *ss*-methylene ooo o-tetraethyl di(phosphorodithioate). The empirical and structural formulae and the molecular mass of ethion are as given below:

Empirical Formula	*Structural Formula*	*Molecular Mass*
$C_9H_{22}O_4P_2S_4$		384·5

0.4 In the preparation of this standard, due consideration has been given to the provisions of the *Insecticides Act*, 1968, and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS:2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

---

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for ethion, technical.

*Rules for rounding off numerical values (*revised*).

2. REQUIREMENTS

2.1 Description — The material shall be an amber liquid, free from extraneous impurities or added modifying agents.

2.2 The material shall also comply whith the requirements specified in Table 1.

TABLE 1 REQUIREMENTS FOR ETHION, TECHNICAL

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Appendix	Cl No. of IS: 6940-1982*
(1)	(2)	(3)	(4)	(5)
i)	Ethion content, percent by mass, *Min*	92·0	A	—
ii)	Acidity (as H_2SO_4), percent by mass, *Max*	0·30	—	11·3
iii)	Moisture content, percent by mass, *Max*	0·20	—	4

*Methods of tests for pesticides and their formulations (*first revision*).

3. PACKING AND MARKING

3.1 Packing — The material shall be packed as per requirements given in IS : 8190 (Part II)-1980* as applicable to packing of malathion technical.

3.2 Marking — The containers shall bear legibly and indelibly the following information in addition to any other information as is necessary under the *Insecticides Act* and Rules framed thereunder:

a) Name of the material;

b) Name of the manufacturer;

c) Date of manufacture;

d) Batch number;

e) Ethion content, percent (*m/m*);

f) Net mass of the contents; and

g) A cautionary notice as worded in Insecticides Act and Rules.

*Requirements for packing of pesticides, Part II Liquid pesticides (*first revision*).

3.2.1 Each container may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in 'Indian Standard methods for sampling of pesticides and their formulations' (*under preparation*).

NOTE — Till such time the standard under preparation is published, the samples shall be drawn as agreed to between the parties concerned.

5. TESTS

5.1 Tests shall be carried out by appropriate methods referred to in col 4 and 5 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

[*Table* 1, *Item* (i)]

DETERMINATION OF ETHION CONTENT

A-0. GENERAL

A-0.1 For the determination of ethion content, two methods namely infra-red spectrophotometric method (*see* **A-1**) and column chromatography followed by phosphate determination as quinoline phosphomolybdate (*see* **A-2**) have been specified. Either of the two methods may be used for the determination of ethion content but infra-red spectophotometric method shall be the referee method.

*Specification for water for general laboratory use (*second revision*).

A-1. INFRA-RED SPECTROPHOTOMETRIC METHOD

A-1.1 Method — The method consists of dissolving ethion in tetrachloroethylene, spectroscopic grade; scanning the solution in the wave length region of 9·5-10·5μ (1050-950 cm^{-1}) in an infra-red spectrophotometer and measuring the characteristic absorption peak at 9·88μ (1012 cm^{-1}) through the base point at 10·15μ (985 cm^{-1}).

NOTE — Since compounds with ethoxy groups attached to phosphorus exhibit strong absorption band between 1030 and 1010 cm^{-1} and the absorption band is attributed to P-O(C) vibration in the molecule, a wide range spectrum for identification of individual peaks may be taken.

A-1.2 Apparatus

A-1.2.1 *Infra-Red Spectrophotometer* — Capable of recording in the region of 2000 to 400 cm^{-1}. The slit width, gain, response time and scanning speed should be so adjusted as to produce a satisfactory signal to noise ratio and an adequate resolution. For the determination of ethion content by this method following parameters have been observed to be suitable:

Slit programme	1 000
Gain	4
Attenuator speed	1 100
Scan time	100 cm^{-1}/min
Suppression	5
Scale	IX
Source current	0·3 A

A-1.2.2 *Absorption Cells* — sealed absorption cells with sodium chloride or potassium bromide windows having an internal light path 0·1 mm.

A-1.2.3 *Hypodermic Glass Syringe* — 1·0 or 2·0-ml capacity, fitted with an 1·25 mm (18 gauge) slip on type needle.

A-1.2.4 *Volumetric Flasks* — capacity 25-ml, glass stoppered.

A-1.3 Reagents

A-1.3.1 *Ethion* — of known (plus 99 percent) purity.

A-1.3.2 *Tetrachloroethylene* — spectroscopic grade.

A-1.4 Procedure

A-1.4.1 *Preparation of calibration graph*

A-1.4.1.1 Weigh accurately into separate 25-ml volumetric flasks 50, 75, 100, 125 and 150 mg of pure ethion. Dissolve in tetrachloroethylene and dilute to the mark after mixing thoroughly.

A-1.4.1.2 Fill the absorption cell with each of the five standard solutions of ethion (*see* **A-1.4.1.1**) in turn, starting with the dilute standard solution, by means of hypodermic syringe. Make three scans of each of these solutions in the wavelength region 9·5—10·5μ (1050 to 950 cm^{-1}).

A-1.4.1.3 For each of the scans of the standard solutions, measure the absorbance at 1012 cm^{-1} and the base point optical absorbance at 985 cm^{-1}. The difference between the maximum absorbance at 1012 cm^{-1} and the base point optical absorbance at 985 cm^{-1} gives absorbance due to ethion.

A-1.4.1.4 Average the absorbance of the three scans of each of the standard solution (*see* **A-1.4.1.1**) and plot a graph, taking absorbance as ordinate against concentration as abscissa, for the five calibration solutions.

A-1.4.2 *Estimation of Ethion Content*

A-1.4.2.1 Weigh accurately a quantity of the sample containing 100-150 mg of ethion in a 25-ml volumetric flask and proceed as given in **A-1.4.1.1** to **A-1.4.1.3**.

A-1.5 Calculation

$$\text{Ethion content, percent by mass} = \frac{M \times 100}{M_1}$$

where

M = mass, in mg, of ethion as calculated from the calibration curve.

M_1 = mass, in mg, of sample taken for test.

A-2. COLUMN CHROMATOGRAPHY/GRAVIMETRIC METHOD

A-2.1 Method — Ethion is separated from impurities by column chromatography and an aliquot of purified ethion made to volume is decomposed with a mixture of sulphuric acid and nitric acid. The phosphate thus obtained is precipitated as quinoline phosphomolybdate and is estimated gravimetrically.

A-2.2 Apparatus

A-2.2.1 *Chromatographic Column* — about 40 cms in length and having 20 mm internal diameter, with a fritted glass disc (G 3) at the bottom that supports the absorbant and a burette tap. The top shall have a B 19 joint fused.

A-2.2.2 *Sintered Glass Crucible* — porosity G-4, 30-ml capacity.

A-2.3 Reagents

A-2.3.1 *Silica Gel* — Chromatographic grade (60-120 mesh), dried for 4 hours at 110°C.

A-2.3.2 *Ether-Hexane Mixture* — 10 percent ether in hexane (*v/v*).

A-2.3.3 *Sulphuric Acid* — analytical reagent grade.

A-2.3.4 *Nitric Acid* — analytical reagent grade.

A-2.3.5 *Citric Molybdate Solution* — prepared as given in A-2.3.5.1 and A-2.3.5.2.

A-2.3.5.1 Weigh 54 g of molybdic anhydride (MoO_3) in a 500-ml beaker. Add 200 ml of distilled water and 11 g of sodium hydroxide pellets. Heat the beaker until molybdic anhydride dissolves.

A-2.3.5.2 Dissolve 60 g of citric acid crystals in 250 ml of water in a one litre beaker and add 140 ml of hydrochloric acid (relative density 1·16). To this solution, add solution prepared in A-2.3.5.1 with stirring. Filter and dilute to one litre. This solution may be green or blue in colour. Add 0·5 to 1 percent solution of potassium bromide until green colour disappears. Keep solution in a dark place in a well stoppered bottle of polyethylene.

A-2.3.6 *Quinoline Solution* — dilute 60 ml of concentrated hydrochloric acid with 300-400 ml water in a beaker and heat to 70 to 80°C. Slowly add 50 ml of pure quinoline stirring all the while when quinoline dissolves. Cool the solution, dilute with distilled water and filter into a one litre volumetric flask and make up the volume with water. Keep solution in a polyethylene bottle.

A-2.4 Procedure

A-2.4.1 Weigh about 22·5 g of silica gel at room temperature into a 250-ml beaker and make a slurry with hexane. Mix the slurry with a glass rod and transfer to the column by keeping the stop-cock open and without allowing the gel to dry during the operation by using more hexane. Gently tap the column to drive out any air bubbles entrapped. Apply nitrogen pressure 0·7 Kg/cm^2 (10 lbs) on the column for compaction of the gel. Do not allow the liquid level to drop below the top of the silica gel column. Layer about 2 cms of washed sand on the top of the column to prevent disturbing the silica gel while charging the solvent or sample.

A-2.4.2 Accurately weigh a sample containing 0·5 g of ethion technical in a 25-ml beaker and dissolve in about 2-3 ml of hexane. When the liquid level is about 1 mm above the silica gel, pour the solution of ethion

carefully along the sides of the column. Rinse the beaker and the column with three times 2 ml of hexane. Again, do not allow the liquid level to fall below the top of the silica gel column.

A-2.4.3 Elute with 250 ml of 10 percent ether in hexane at a rate of 5 ml/min. Discard the first 50 ml fraction and collect the next 150 ml fraction in a cylinder. Transfer this fraction into a 250-ml volumetric flask quantitatively using hexane and make up to the mark.

A-2.4.4 Pipette out 25 ml aliquot (*see* A-2.4.3) into a dry Kjeldahl flask of 100-ml capacity. Evaporate the solvent on a water bath. Cool the flask and add carefully 4·5 ml of concentrated sulphuric acid and 1·5 ml concentrated nitric acid. Heat on a low flame till dense fumes appear. Let it cool and repeat the operation. Add 1 ml nitric acid and continue heating till the solution becomes perfectly clear and almost colourless.

A-2.4.5 Transfer the contents quantitatively, on cooling, into a 500-ml beaker with water to a volume of about 25-30 ml. Boil for 5 minutes and dilute with water to about 150 ml. Add 25 ml of citric molybdate solution and boil for 3 minutes. Immediately add 12·5 ml of quinoline solution from a burette. The quinoline solution should be added dropwise and the contents of the beaker must be stirred continuously so that the precipitate obtained can be easily filtered. Let the beaker stand for 5 minutes stirring for 5-10 seconds each time and then cool it to room temperature.

A-2.4.6 Filter by suction through a G4 sintered glass crucible. Wash precipitate four times by decantation and then wash with plenty of distilled water. Dry the precipitate at 110°C for 2 hours.

A-2.4.7 Weigh the precipitate as quinoline phosphomolybdate $(C_9H_7N)_3H_3PO_4.12\ MoO_3$.

A-2.5 Calculation

Ethion content, percent by mass

$$= \frac{384{\cdot}5}{2 \times 31} \times 0{\cdot}01399 \times \frac{m}{M} \times \frac{250}{25} \times 100$$

$$= \frac{86{\cdot}76 \times m}{M}$$

where

m = mass, in g, of the precipitate obtained; and

M = mass, in g, of the sample taken for test.

NOTE — 0·01399 is the conversion factor for quinoline phosphomolybdate to phosphorus as P.

(*Continued from page* 2)

Members	*Representing*
SHRI I. NARSAREDDY	Department of Agriculture, Government of Andhra Pradesh, Hyderabad
SHRI C. DHARMA RAO (*Alternate*)	
SHRI G. NATARAJAN	Indofil Chemicals Limited, Bombay
DR V. N. NIGAM	Ministry of Defence (DGI)
SHRI P. N. AGARWAL (*Alternate*)	
SHRI M. M. PADALIA	Department of Agriculture, Government of Gujarat, Ahmadabad
SHRI R. G. JADEJA (*Alternate*)	
DR S. Y. PANDIT	Bayer (India) Limited, Bombay
SHRI C. G. DHARNE (*Alternate*)	
DR P. S. PHADKE	Sandoz (India) Limited, Thane
SHRI A. V. NATU (*Alternate*)	
SHRI S. M. PRADHAN	Metal Containers Sectional Committee, MCPD 12, ISI
SHRI Y. A. PRADHAN	Rallis India Limited, Bombay
DR Z. J. KAPADIA (*Alternate*)	
SHRI D. N. V. RAO	Tata Chemicals Limited, Mithapur
SHRI C. NEELKANTAN (*Alternate*)	
REPRESENTATIVE	Pesticides Association of India, New Delhi
SECRETARY	Central Insecticides Board, Directorate of Plant Protection, Quarantine & Storage (Ministry of Agriculture), Faridabad
DR R. C. GUPTA (*Alternate*)	
SHRI A. C. SHROFF	Excel Industries Limited, Bombay
SHRI P. V. KANGO (*Alternate*)	
DR K. S. SINGH	Indian Veterinary Research Institute (ICAR), Izatnagar
SHRI S. C. SRIVASTAVA (*Alternate*)	
SHRI N. S. VENKATARAMAN	Department of Agriculture, Government of Tamil Nadu, Madras
DR R. V. VENKATARATNAM	Regional Research Laboratory (CSIR), Hyderabad
DR NAGABUSHAN RAO (*Alternate*)	
DR R. R. VENKATESWARAN	Union Carbide (India) Limited, New Delhi
DR K. N SHRIVASTAVA (*Alternate*)	
SHRI E. R. VICCAJEE	Paper and Flexible Packaging Sectional Committee, MCPD 14, ISI
SHRI N. G. WAGLE	Pest Control (India) Private Limited, Bombay
SHRI M. R. BAJIKAR (*Alternate*)	
DR B. L. WATTAL	National Institute of Communicable Diseases, Delhi
SHRI G. C. JOSHI (*Alternate*)	
SHRI T. PURNANANDAM, Director (Agri & Food)	Director General, ISI (*Ex-officio Member*)

Secretary

SHRI M. L. KUMAR
Deputy Director (Agri & Food), ISI